Scratch Hardness. II : X-Ray Investigation of Structural Change

Scratch Hardness. II
X-Ray Investigation of Structural Change*

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Synopsis

The structural change due to scratching or indenting aluminium and silver was studied by X-ray analysis, and it was seen that it was a reasonable interpretation that the scratch hardness of a metal is related to the annealed state, which is contrary to Tammann's view.

I. Introduction

From his observation that the change in the scratch width with the increase in cold-working of a metal was very small, Tammann[1] concluded that the scratch hardness was related to the state of maximum hardening, being independent of previous history of the metal. Recently, the relation between load and scratch width was precisely examined with various metal single crystals,[2] and by taking into account the interesting experimental results of Bowden and Ridler[3] it was considered that the scratch hardness was related rather to the annealed state than to the hardened state of a metal. It was also remarked there that the validity of this consideration would be verified probably by X-ray analysis. Hence, the present study was carried out for this purpose and, further, to see the difference between the scratch and the indentation.

II. Experimental method

When the scratch width or the area of indentation is very small, the irradiation of X-ray of these regions is experimentally difficult and, therefore, compared with those used in the previous experiment,[2] heavy loads were used, namely, 500g and 1kg for scratch and 10kg for indentation. Specimens were single- and poly-crystals of aluminium and silver. The effective focus of the X-ray was squeezed by electric lens to about 0.5 mm in diameter. Openings of the first and the second slits were respectively 0.5mm and 0.03mm in diameter; the second slit was carefully made in a lead plate with fine needles, and was of the form of a truncated cone, whose diameters were about 30 μ and 100 μ, respectively. The surface irradiated was directly measured with the result of the order of 0.2 mm in diameter. The

* The 911th report of the Research Institute for Iron, Steel and Other Metals.

(1) G. Tammann, *Lehrbuch der Metallkunde*, (1932).
(2) T. Sutoki and T. Hikage, Sci. Rep. RITU, **A 10** (1958), 85.
(3) F.P. Bowden and K.E.W. Ridler, Proc. Roy. Soc., **154** (1936), 640.

X-ray tube was operated under 25kV and 8mA. The severely cold-rolled specimen was electropolished after mechanical polishing, and the flat surface was scratched with the diamond of Martens' type or indented with Vickers' indentor under the above-mentioned loads. In X-ray analysis, $h^2+k^2+l^2=27$ reflection was used with Cu-target, Bragg angle of which was 76°58′. In some cases, unfiltered X-ray was used.

III. Results of experiment

The specimen holder was so devised that not only the irradiation might be carried out in any direction, but also the specimen might be displaced smoothly in parallel to itself in the direction perpendicular to the primary beam. X-ray photographs were taken at every 0.3mm along the length of 3mm on the surface of the specimen. The structure of aluminium specimen cold-rolled as mentioned above was homogeneous over small irradiated region as shown in Photo. 1. The specimen was then scratched under the load of 1 kg, and the scratch width was about 0.6mm. Typical example of the X-ray photograph reflected from the scratch track is shown in Photo. 2. By comparing Photos. 1 and 2 with each other, it will be seen that line broadening takes place, and that the preferred orientation of crystallites almost disappears, resulting in homogeneous diffuse ring, and that in most cases very sharp diffraction spots are clearly observable on the homogeneous diffuse ring, which is not shown in the X-ray photograph of the original state. Similar experiments were carried out in the case of indentation with the same specimen, and Photo. 3 shows an example of the results, the load being 5 kg. As shown in Photo. 3, in the case of indentation the Debye ring diffused, and the effect of preferred orientation of crystallites disappeared almost perfectly, but the spotty diffraction patern could not be observed.

Further, a similar experiment was carried out with a single crystal of aluminium. Photo. 4 shows the original state of the specimen. When it was scratched under the load of 500g, the scratch width was of the order of 0.4mm. To see the effect of scraching on the surrounding matrix, X-ray photographs were taken at every 0.15 mm from the centre of the scrach track to the edge of the surface. Photo. 5 is the photograph of the region about 0.3mm from the edge of a scratch track, which shows the diffusion of Laue spots due to strain. In the previous experiment[2] the hardness suddenly fell at a distance of about 30μ from the edge of the track, and then gradually decreased to the value of the annealed state. The present result of X-ray examination is also explained by the existence of the strained state in the vicinity of the scratch track. In the photograph taken of the scratch track, Laue spots, though poor, was observable on the Debye ring, as shown in Photos. 6 a and 6 b, because of the specimen being somewhat annealed.

IV. Discussion of the results

By comparing the X-ray diffraction patterns of the scratch track and the original surface of a cold-worked polycrystalline metal with each other, important changes in the diffraction pattern due to the scratching will be seen, that is, (1)

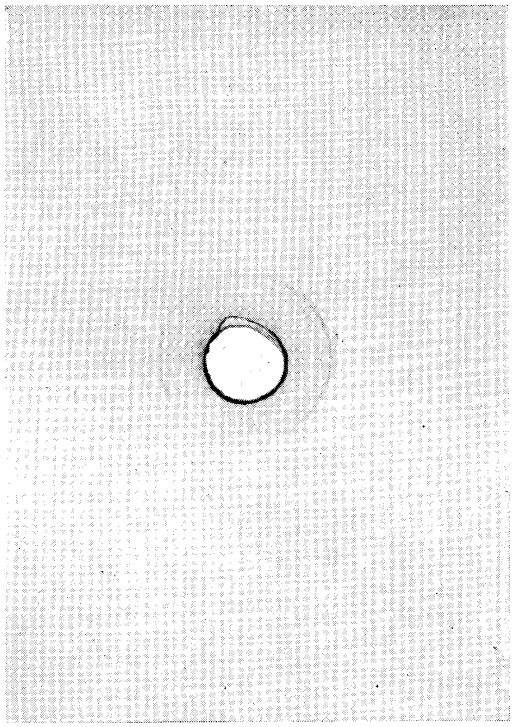

Photo. 1
X-ray photograph of the original state of the specimen of aluminium.
Irradiated area 0.2mm in dia.

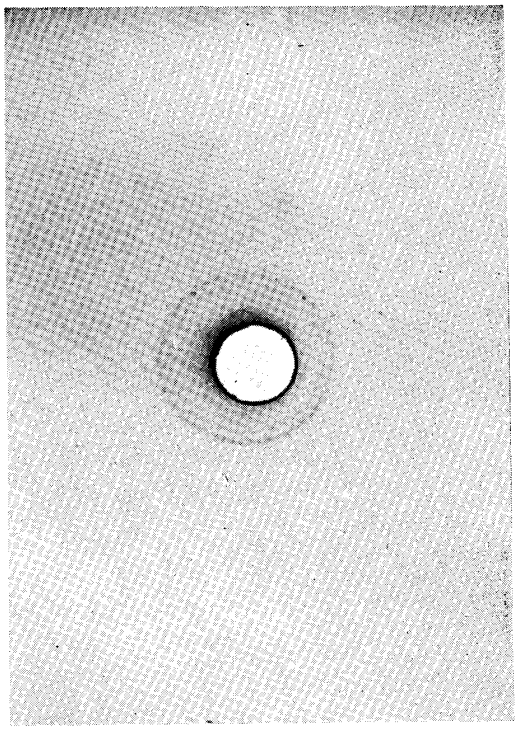

Photo. 2
X-ray photograph of the scratch track of the specimen.

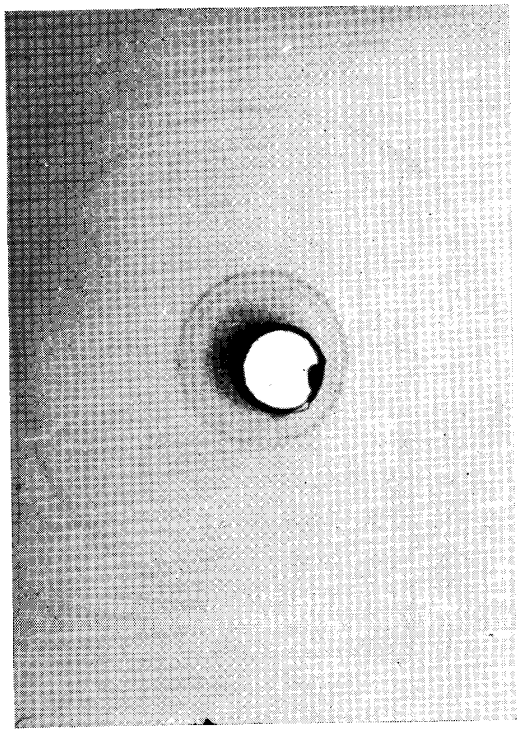

Photo. 3
X-ray photograph of the region indented.

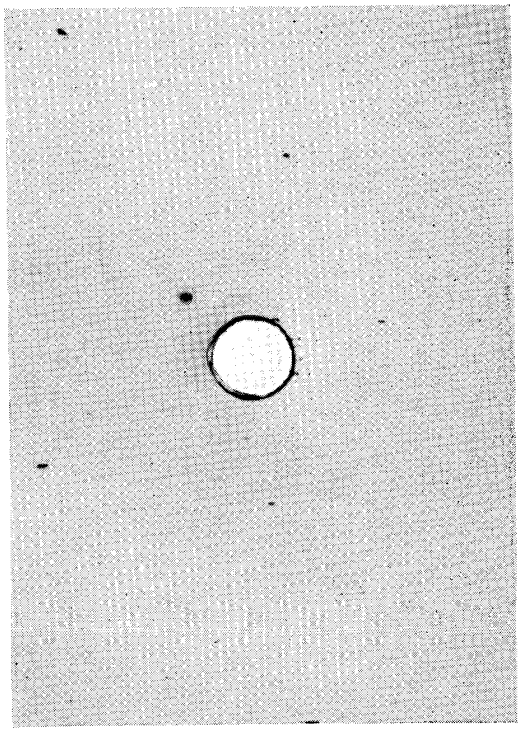

Photo. 4
Back reflection Laue photograph of the original state of the specimen of aluminm single crystal.

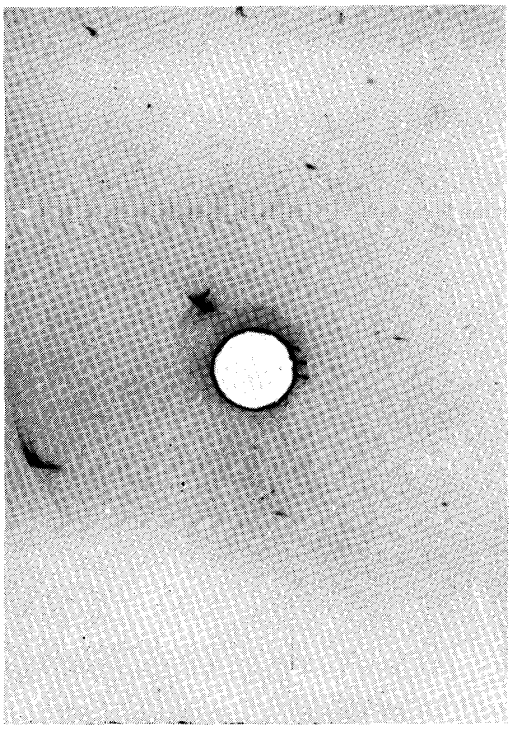

Photo. 5
Back reflection Laue photograph of the vicinity of the scratch track.

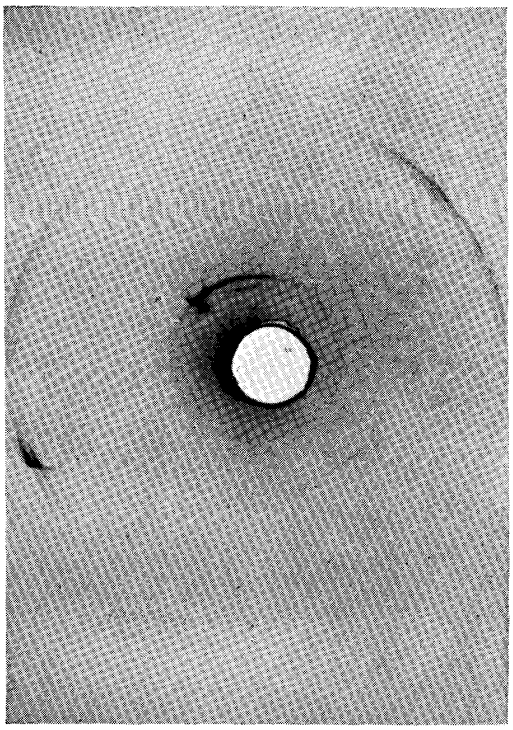

Photo. 6 a
X-ray photograph of the scratch track. Unfiltered

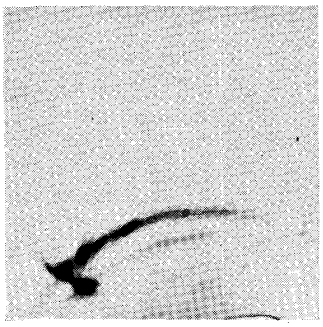

Photo. 6 b Enlarged.

the formation of homogeneous diffuse ring; (2) the disappearance of preferred orientation of crystallites; (3) the appearance of a few Laue spots on the homogeneous diffuse ring. The X-ray photographs taken of the indented part were generally of homogeneous diffuse ring as shown in Photo. 3, showing severe cold-working by indentation. The spotty diffraction pattern mentioned above, however, could not be obtained in the case of indentation. This result will show that the mechanism of scratch is different from that of the indentation, that is, the spotty diffraction pattern may be explained to be caused by the recrystallization in the thin layer of scratch track due to heat evolved. The half penetrating depth of Cu-K_α radiation is calculated to be about 60μ for alumininm and, therefore, the above results show the structural change within a thin layer of the surface. From these it may be concluded that scratch hardness is related to the annealed

state irrespective of a previous history of a metal. On the other hand, it has been reported by some workers[4] that aluminium, in some cases, recrystallizes at room temperature after severe cold-working and, in fact, the recrystallization was often obsereved at room temperature in aluminium cold-rolled about 95 per cent in the present experiments and, therefore, the alternative interpretation may also be possible in this case. Hence, a similar experiment was carried out with silver. Photos. $7 \sim 10$ show the results in the case of cold-rolled silver. Photo. 7 is the diffraction photograph after electro-polishing, and Photo. 8 is that taken of the mechanically polished surface. The photographs taken after scratching or indenting the electro-polished surface are shown respectively in Photos. 9 and 10. From these results it will be seen that the strained state due to indentation corresponds to the state mechanically polished, and the half penetrating depth of Cu-K_α radiation was calculated to be about 3μ for silver. On the other hand, in the case of scratching the diffraction spots are clearly observed on the continuous diffuse ring, and are extremely sharp compared with the case of original state shown in Photo. 7.

If the spotty pattern originates actually from partial recrystallization, a similar pattern will also be obtained by annealing a worked specimen under some conditions appropriate to the present case. After some trials, such X-ray photographs as shown in Photo. 11 were always reproducible, that is, the severly cold-worked silver

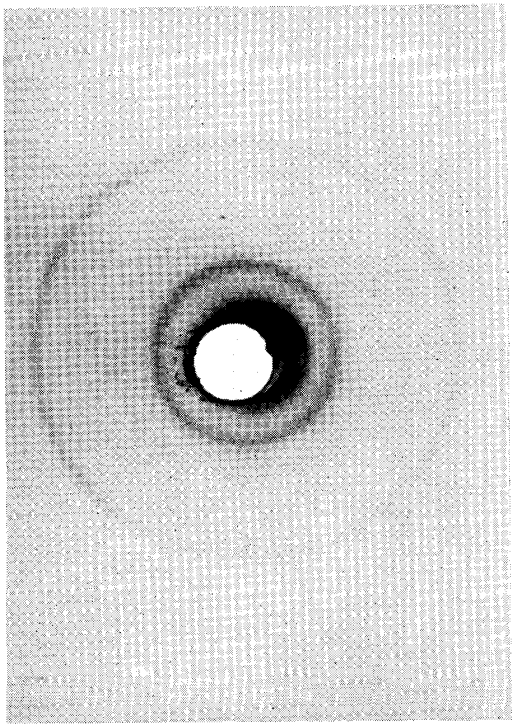

Photo. 7
X-ray photograph of the specimen of silver after mechanical polishing. Beam dia. 0.3mm. Unfiltered.

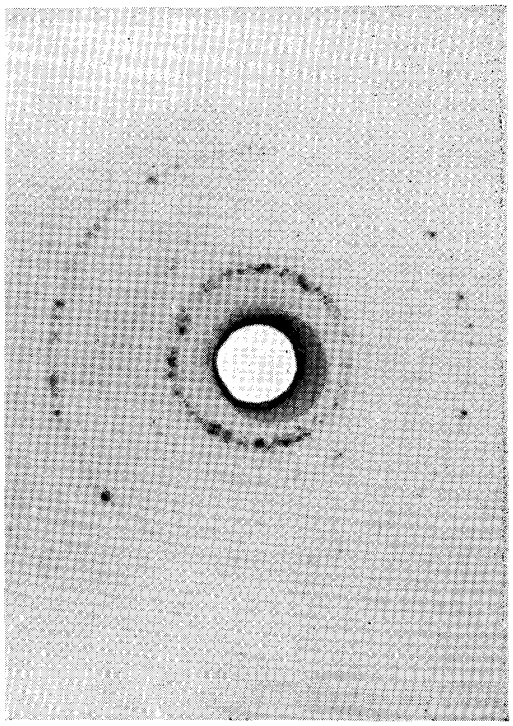

Photo. 8
X-ray photograph of the specimen of silver after electro-polishing. Unfiltered.

(4) A. Kelly and W.T. Roberts, Acta. Met., **3** (155), 96.

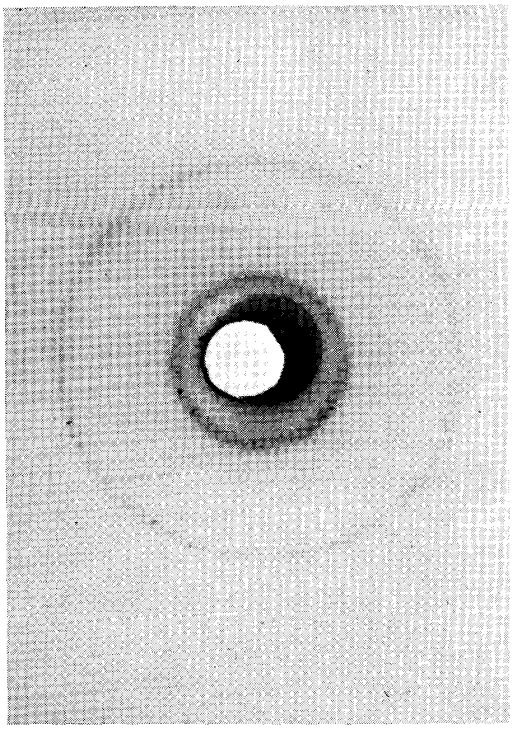

Photo. 9
X-ray photograph from the scratch track. Unfiltered.

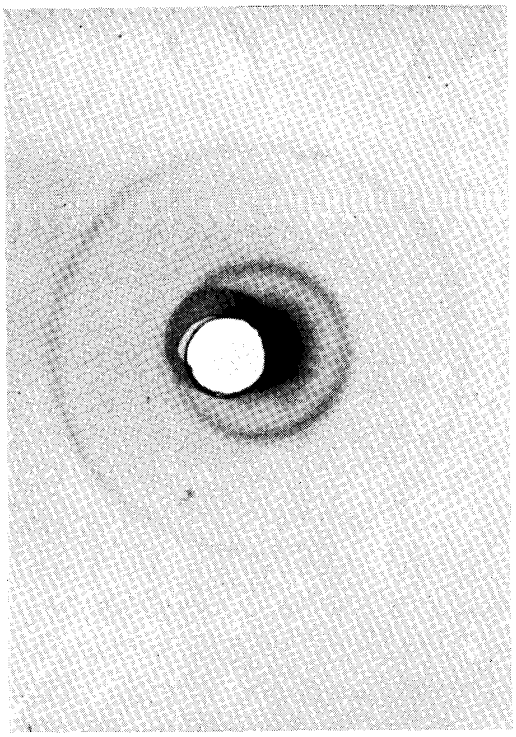

Photo. 10
X-ray photograph from the region indented. Unfiltered

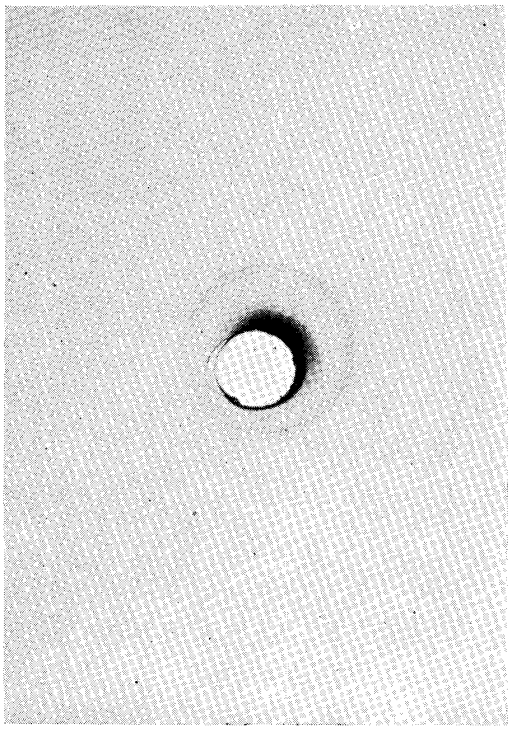

Photo. 11
X-ray photograph of the specimen after instantaneous heating at 400°C

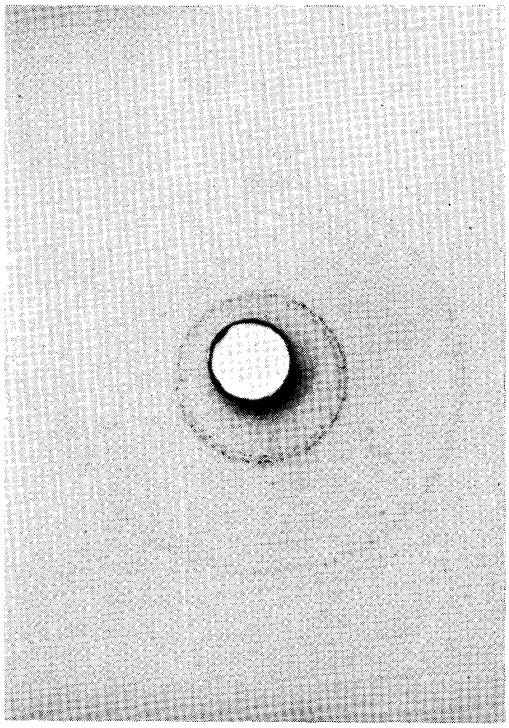

Photo. 12
X-ray photograph of the specimen after instantaneous heating at 500°C

specimen was kept for about 3 seconds in contact with an asbestos plate held at about 400°C in a furnace, and then quenched in water. Some recrystallizations taking place in the surface layer can be seen faintly in the photograph. When the temperature was raised to 500°C, the recrystallizations became clearly observable as shown in Photo. 12. From these results, it may be said that instantaneous rise in local temperature due to scratching is about 400~500°C. Thus, it is confirmed that the scratch hardness of a metal is related rather to the annealed state than to the hardened state as previously reported[2]; in other words, the scratch hardness is incapable of showing the degree of strain-hardening of a metal. Strictly speaking, some effects of cold-working are unavoidable in the hardness of indentation type, and hence, the scratch hardness is superior at least in this respect, provided that the structure of a metal is not mechanically and thermodynamically metastable.

Summary

(1) X-ray study of the structural change due to scratching or indentation was carried out with single crystals and polycrystals of aluminium and silver.
(2) The spotty diffraction pattern on the diffuse ring was observed in the X-ray photographs taken of the scratch track of a severely cold-rolled metal, but not observed in the case of indentation.
(3) The spotty pattern of a scratch track just corresponded to the X-ray photograph taken of partially recrystallized metal.
(4) Differing from the indentation hardness, the scratch hardness is related rather to the annealed state than to the worked state of a metal, being incapable of showing the degree of strain-hardening.

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